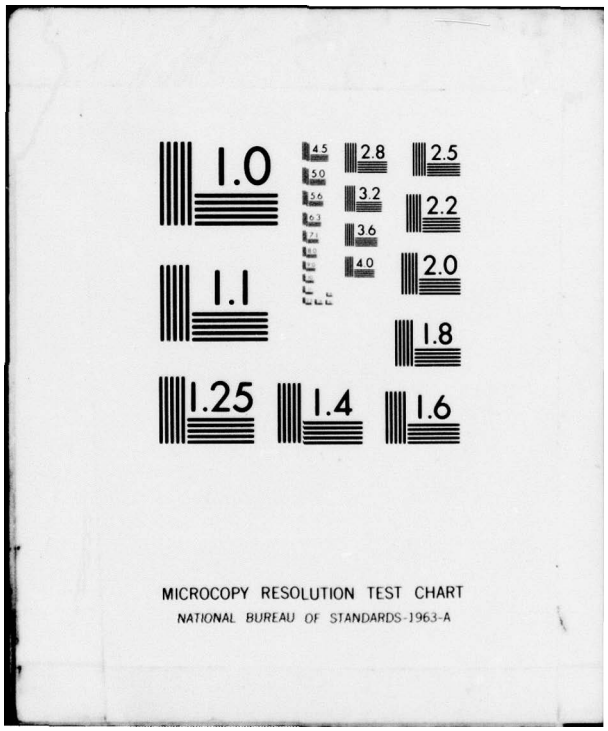
AD-A074 073 SOUTHWEST RESEARCH INST SAN ANTONIO TX ARMY FUELS AN--ETC F/G 11/8
THE PHYSICAL AND CHEMICAL CHARACTERIZATION OF TEN MILITARY TURB--ETC(U)
SEP 79 F M NEWMAN, L L STAVINOHA DAAK70-79-C-0142
UNCLASSIFIED AFLRL-115 NL

| OF |
AD
A074073

END
DATE
FILMED
10-79
DDC


1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
5.6
6.3
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

THE PHYSICAL AND CHEMICAL CHARACTERIZATION OF TEN MILITARY TURBINE ENGINE LUBRICANTS

FINAL REPORT
AFLRL No. 115

by

Frank M. Newman and Leo L. Stavinoha
U.S. Army Fuels and Lubricants Research Laboratory
Southwest Research Institute
San Antonio, Texas

for

U.S. Army Aviation Research and Development Command
St. Louis, Missouri

Under contract to

U.S. Army Mobility Equipment Research and Development Command
Energy and Water Resources Laboratory
Fort Belvoir, Virginia

Contract No. DAAK70-79-C-0142

September 1979

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Trade names cited in this report do not constitute an official endorsement or approval of the use of such commercial hardware or software.

DDC Availability Notice

Qualified requestors may obtain copies of this report from Defense Documentation Center, Cameron Station, Alexandria, Virginia 22314.

Disposition Instructions

Destroy this report when no longer needed. Do not return it to the originator.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: AFLRL No. 115
2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER

4. TITLE *(and Subtitle)*: The Physical and Chemical Characterization of Ten Military Turbine Engine Lubricants

5. TYPE OF REPORT & PERIOD COVERED: Final Report July 1979 — September 1979

6. PERFORMING ORG. REPORT NUMBER: AFLRL No. 115

7. AUTHOR(s): Frank M. Newman, Leo L. Stavinoha

8. CONTRACT OR GRANT NUMBER(s): DAAK70-79-C-0142

9. PERFORMING ORGANIZATION NAME AND ADDRESS: U.S. Army Fuels & Lubricants Res. Lab., P.O. Drawer 28510, San Antonio, TX 78284

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 32 P.

11. CONTROLLING OFFICE NAME AND ADDRESS: U.S. Army Mobility Equipment Research & Development Command, Attn: DRDME-GL, Fort Belvoir, VA 22060

12. REPORT DATE: September 1979

13. NUMBER OF PAGES: 25

14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*: AFLRL-115

15. SECURITY CLASS. *(of this report)*: Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*: Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Turbine Engine Lubricant	Diester	Synthetic Lubricants
Lubricant	Polyol Ester	Chemical Analysis
Base Stock	Synthetic Oils	
Base Stock Composition	Esters	

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Four MIL-L-23699B and six MIL-L-7808G turbine engine lubricants have been characterized from the physical properties and chemical composition of their basestocks. The four MIL-L-23699 lubricants were found to be 100% polyol esters. Of the six MIL-L-7808 lubricants, four were mixtures of polyol esters and diesters, one was 100% polyol ester, and one was 100% diester. Correlation of the chemical data is made to some of the physical properties wherever possible.

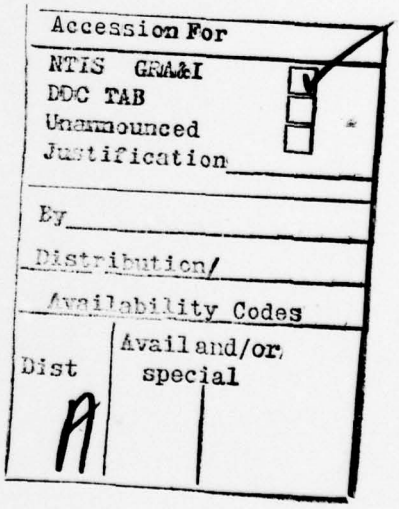
DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

387 339

TABLE OF CONTENTS



LIST OF TABLES

LIST OF FIGURES

FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL), located at Southwest Research Institute, San Antonio, Texas under Contract DAAK70-79-C-0142 during the period July 1979 through September 1979. The contracting officer representative was Mr. F.W. Schaekel at DRDME-GL, USAMERADCOM, Ft. Belvoir, Virginia.

ACKNOWLEDGEMENT

The authors acknowledge the helpful suggestions and assistance provided by AFLRL staff members. The encouragement of Mr. Edward J. Hollman (DRDAV-QP, AVRADCOM), Dr. Erwin V. Zaretsky (NASA/Lewis), and Mr. Maurice E. LePera (DRDME-GL,MERADCOM) is also acknowledged.

I. INTRODUCTION AND BACKGROUND

Analytical methods for determining the composition of new and used synthetic lubricants have been developed as an important part of the Army's on-going lubrication research and development effort.[1-5]* A logical approach for the systematic analysis of lubricants has been applied to a wide variety of selected military and commercial lubricants. Hybrid lubricant blends of synthetic hydrocarbons, esters, and mineral oils have been separated and analyzed through the combined use of spectroscopy and chromatography.[1,2] Ester fractions have been further evaluated by chemical hydrolysis and derivitized to determine the exact composition of the parent acid and alcohol components. While characterization of lubricants to include more detailed additive composition is not yet as complete as would be desired, the analytical approaches presently employed provide compositional information previously unattainable.

Only in the last several years has the capability existed to determine finished mixed-ester lubricant original basestock composition. Lubricant composition has been previously accepted based on the general information supplied by the manufacturer. In many cases, the precise chemical composition has not been revealed by or has not been known by the manufacturer or the end-user. The lack of detailed information prevents user decisions based on composition.

Typically, a lubricant is chosen based on the specification by which it is qualified. Frequently, the specification contains the performance requirements for the lubricant. In field applications or in performance studies the lubricant is thus selected based on the specification. In other cases, a lubricant is selected because it is classified as a lubricant for a given application. Seldom is the composition of the lubricant considered in its overall application selection. Comparison of the composition of different lubricants and correlation of lubricant performance as related to the chemical composition is difficult because

---

* Superscript numbers in parentheses refer to the list of references at the end of this report.

insufficient specific lubricant composition information is available. As a result of the recent developments at the AFLRL, in which lubricants can now be characterized as to their chemical composition, this study was undertaken to define the composition of the base stock materials so that ultimately better correlations can be made. In the work described in this report, the composition of ten lubricant basestocks has been determined in support of a separate effort to better correlate the lubricants' performance in special elastohydrodynamic test equipment.

II. PROCEDURE

Each lubricant was first analyzed by infrared spectroscopy to determine if it was indeed an ester type lubricant and if it contained any mineral oil diluent which might affect the analytical procedures to follow. Physical properties of the lubricants such as the kinematic viscosity at both 40° and 100°C, the flash point, pour point, and API gravity were determined by standard ASTM Methods[6] as required both in the specifications and accepted in all lubricant analysis circles.

Each sample was analyzed by gas chromatography to determined the boiling point distribution of the sample. From the fingerprint chromatograms[4] and the boiling point distribution chromatograms,[1,2] preliminary decisions could be made concerning the lubricants' compositional basestock types. Based on these preliminary decisions, subsequent chemical analyses could be outlined which would ellucidate the chemical structure of each lubricant.

Each lubricant was subjected to alkali hydrolysis, a cleaving process which chemically separates the ester compounds into their original alcohol and acid components. This cleavage process produces the acid as a water-soluble salt which allows the extraction and recovery of the alcohol from the acid salt by organic solvent extraction. In turn, the acid salt can be converted to the free acid which is recovered by a second extraction process. By following this procedure on each sample, the alcohol and acid components are physically separated from each other and can be further treated both chemically and physically to determine the individual members of each family of compounds.

Such treatment involves concentration of the material in organic solvent so that adequate sensitivity could be obtained in the subsequent analyses. The fractions were directly analyzed by gas chromatography and chemically reacted with known reagents to produce derivative compounds which can be readily identified by a second gas chromatographic analysis. The gas chromatographic analyses of the derivatized fractions and the underivatized fractions serve to complement each other and support the interpretation of the results. Quantitative analytical data can be obtained from the gas chromatographic analyses of the derivatized fractions.

Following complete reduction of all chromatographic data, the results were prepared and summarized in both tabular and graphical form. These data illustrate the significant differences and similarities of the lubricant basestocks.

III. EXPERIMENTAL

Except for minor modifications, the experimental procedures have been previously described in References 1 and 2.

A. Hydrolosis Procedure--Hydrolysis was accomplished using potassium hydroxide (50 g) in ethanol (250 ml) and water (50 ml) mixed with 10 to 12 grams of lubricant. This mixture was refluxed for 2 hours to yield the esters' carboxylic acid together with the alcoholic fraction. The alcohols and neutral components were extracted with diethyl ether from the basic reaction mixture, after which the carboxylic acid salt was acidified by a mineral acid (HCl). The carboxylic acid component was then extracted with diethyl ether. All extractions were performed on NaCl saturated solutions to increase recovery.

B. Parent Acid/Alcohol Determination--Acids and alcohols from dibasic esters and alcohols from polyol esters were prepared for gas chromatographic analysis by derivatization. An approximately weighed 0.01-gram sample was placed in a dry 3-ml reaction vial and sealed

using inert disks in the reaction vial caps. A sealed 1-ml glass ampule of a commercially available form of N, O-Bis-(trimethylsilyl)-acetamide in pyridine was opened, poured into the sample vial, shaken for 30 seconds, heated in 60°C (140°F) water for 30 minutes, and then analyzed by gas chromatography. The gas chromatographic peaks attributed to the alcohols and acids were then normalized to 100 percent. Direct carboxylic acid analysis, as well as derivatization, was used for acids from polyol esters. Polyols from polyol esters were also identified by valerate analysis.(7)

C. Gas Chromatography--Two gas chromatographic systems have been developed for high boiling samples such as lubricants. Both methods are essentially ASTM D 2887 *boiling point distribution*(8) techniques with the addition of internal standard, dilution with carbon disulfide, and appropriate data reduction to provide for residue values. These methods have been described in References 1 and 2. The fingerprint technique is described in Reference 4 and uses a polar (OV-17) packed column.

IV. RESULTS AND DISCUSSION

All of the analytical results are presented in tabular form for each of the ten test lubricants in Tables A-1 though A-10 in the Appendix. The compositional data does not consider the additives which might be present in the lubricant and relates only to the basestock of the lubricant. A summary table of the data for the ten lubricants including military specification number is presented in Table 1. This table does not provide the detailed chemical basestock characterization found in the Appendix, but does provide the chemical basestock type in terms of percent dibasic acid ester and polyol ester. Table 1 also summarizes the following properties:

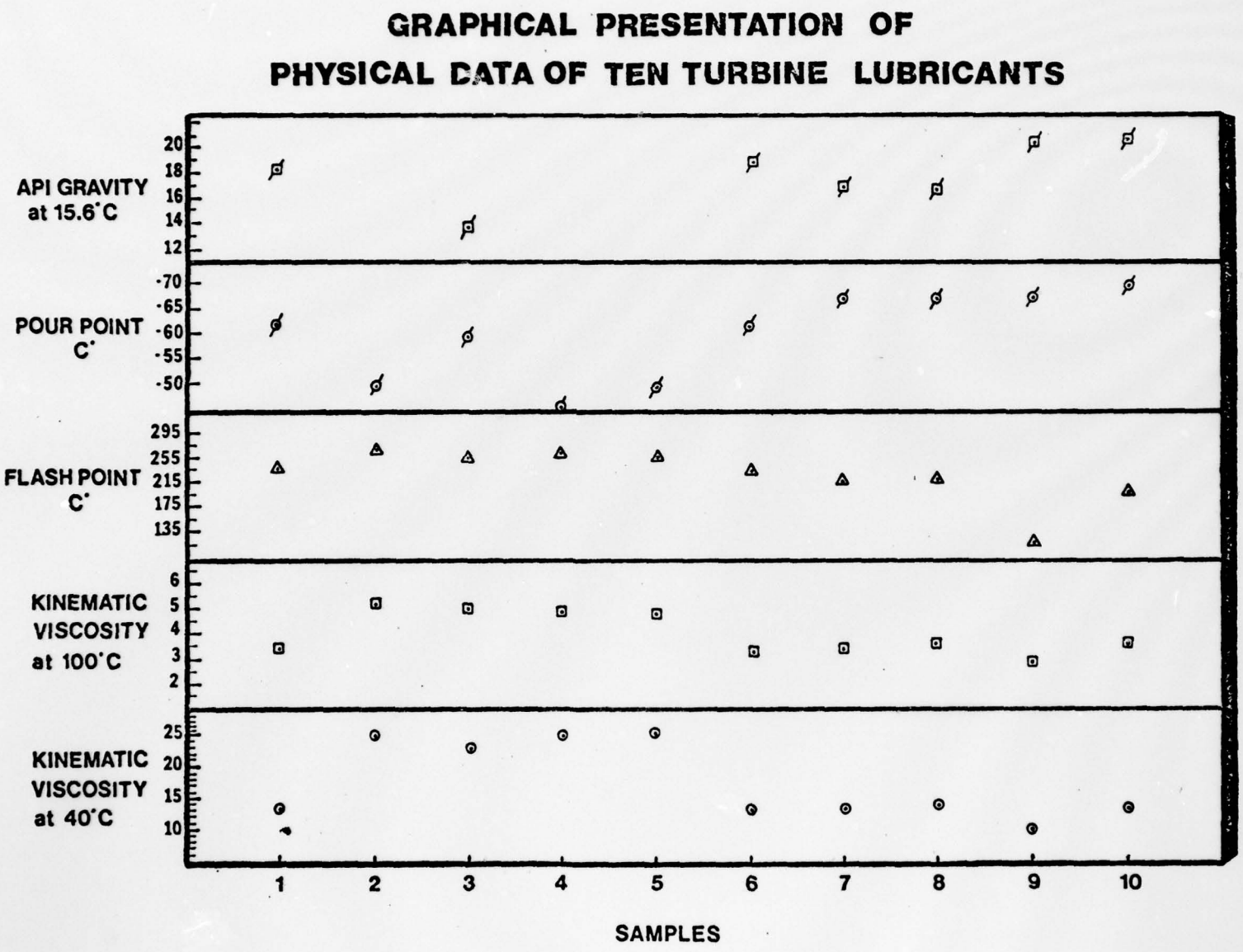
- Kinematic Viscosity, cSt at 98.9°C and 37.8°C,

TABLE 1. SUMMARY OF DATA FOR TEN TURBINE ENGINE LUBRICANTS

Lubricant Sample No.	Type Mil Spec. No.	Chemical Type, %		Kinematic Viscosity, cSt		Flash Point, °C	Pour Point, °C	Gravity °API
		Diester	Polyol Ester	@ 40°C	@ 100°C			
1	7808G	60	40	13.12	3.41	233	-62	18.3
2	23699B	0	100	24.99	5.17	263	-48	*
3	23699B	0	100	23.84	4.99	249	-58	13.4
4	23699B	0	100	24.70	4.98	256	-43	*
5	23699B	0	100	25.03	5.00	254	-48	*
6	7808G	60	40	12.96	3.35	228	-61	18.5
7	7808G	0	100	12.58	3.28	211	-67	16.7
8	7808G	40	60	13.34	3.47	216	-66	16.4
9	7808G	50	50	9.17	2.74	116	-67	21.4
10	7808G	100	0	12.76	3.54	194	-68	20.5

* Insufficient sample

FIGURE 1.


GRAPHICAL PRESENTATION OF
PHYSICAL DATA OF TEN TURBINE LUBRICANTS
API GRAVITY at 15.6°C
20
18
16
14
12
POUR POINT C°
-70
-65
-60
-55
-50
FLASH POINT C°
295
255
215
175
135
KINEMATIC VISCOSITY at 100°C
6
5
4
3
2
KINEMATIC VISCOSITY at 40°C
25
20
15
10
1
2
3
4
5
6
7
8
9
10
SAMPLES

- Flash Point,
- Pour Point, and
- Gravity.

Additional properties in the Appendix include:

- Viscosity Index
- Acid number, and
- Base number

The physical data for the ten lubricants are presented in graphical form in Figure 1. This figure includes the viscosity, flash point, pour point, and API gravity data. The boiling point distribution data are presented in graphical form in Figure 2. The boiling point distribution profiles in Figure 2 emphasize the similarities and the differences between the basestocks of the lubricants.

The chemical composition data for the test lubricant basestocks (Tables A-1 through A-10) have also been presented graphically in Figure 3 as a daisy graph. A daisy graph is a method for representing a large number of parameters or variables in a simple fashion for easy comparision. The turbine engine oil daisy graph key is provided in Figure 4. The angular position of the radial line is characteristic for each individual component. While not necessary, different colors have been used to illustrate the different chemical families of compounds for ease in comparing the composition of the lubricants. Red represents the mono-carboxylic acids present in polyol esters and blue represents the polyol base for the polyol ester. Green represents the mono-alcohols of dibasic acid esters, and black represents the base dicarboxylic acid of the dibasic acid ester. The length of the daisy graph lines in Figure 3 is proportional to the concentration of each component.

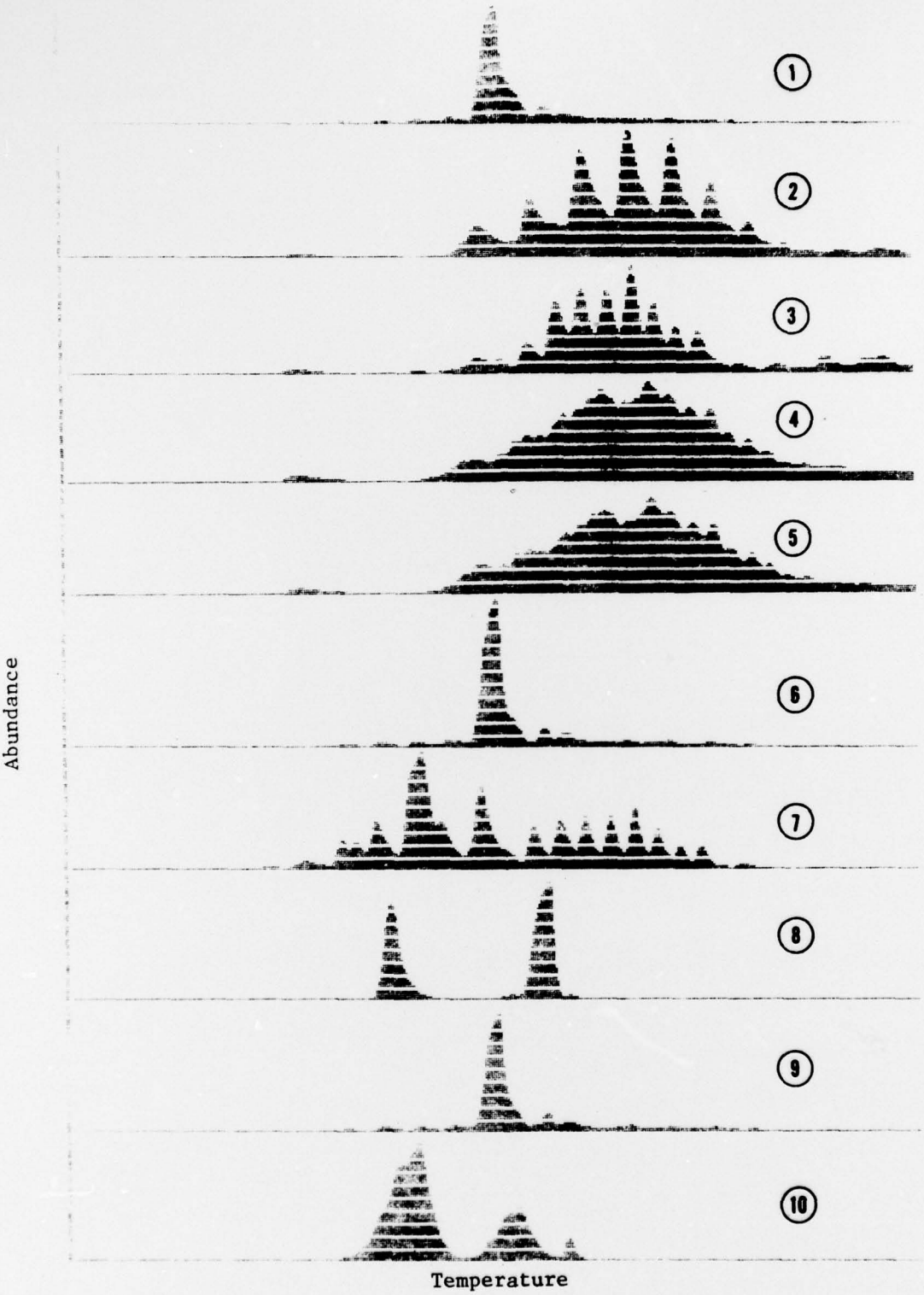


FIGURE 2.
BOILING POINT DISTRIBUTIONS OF TEN TURBINE ENGINE OILS

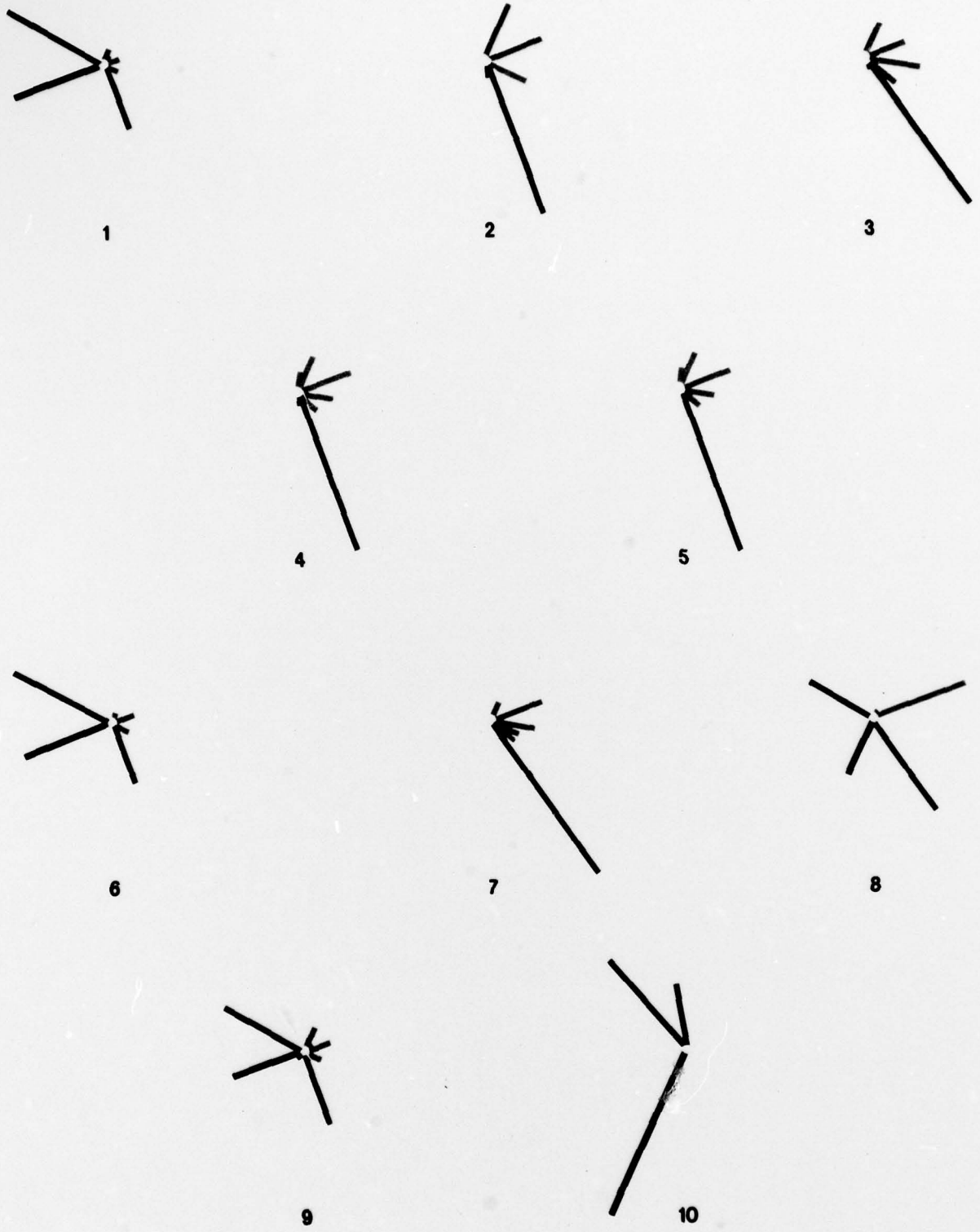


FIGURE 3. DAISY GRAPHS OF COMPOSITIONS OF TEN TURBINE ENGINE OILS

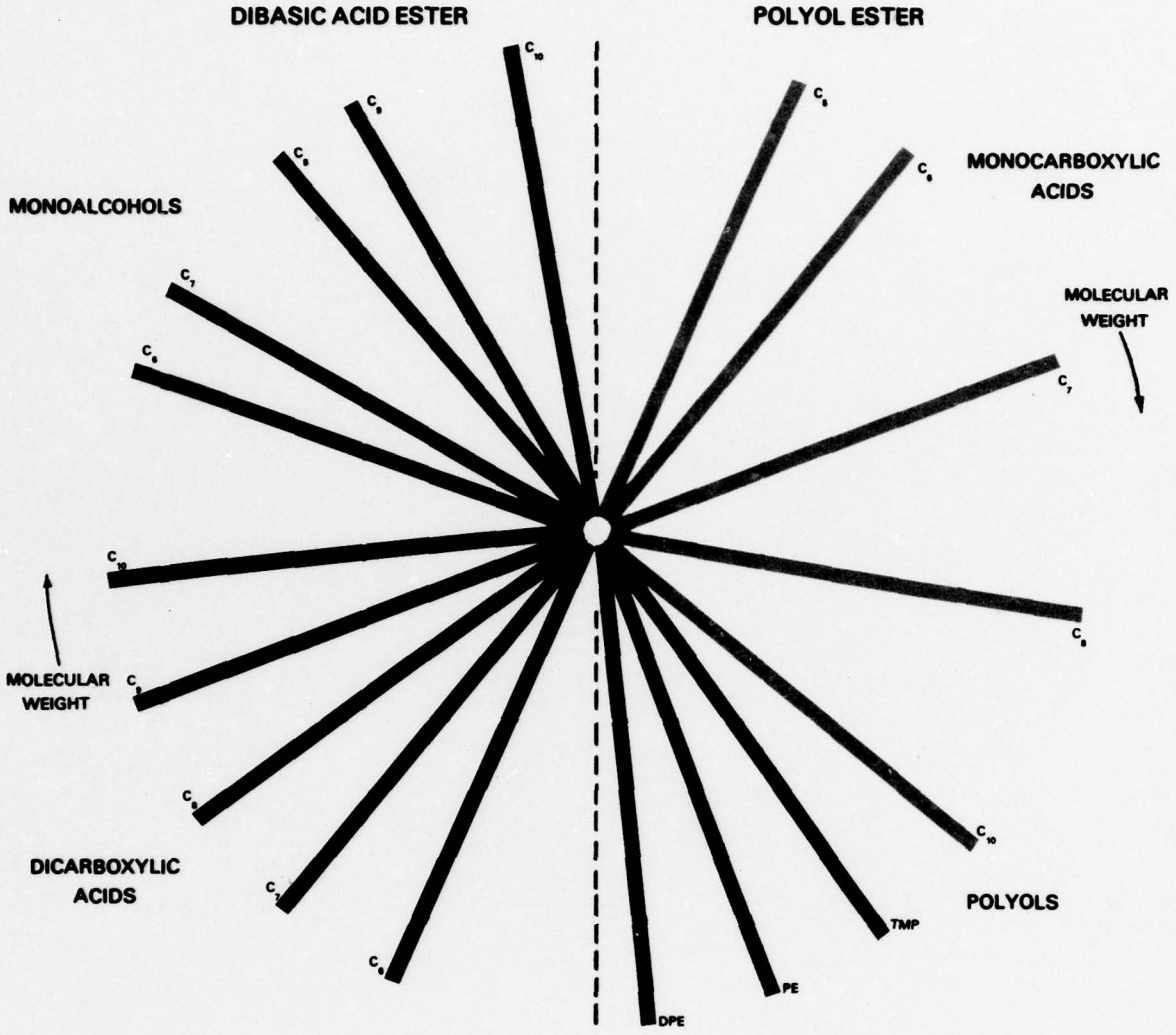


FIGURE 4. TURBINE ENGINE OIL DAISY GRAPH KEY

It can be seen from the boiling point distribution data in Figure 2, that the MIL-L-23699 lubricants, samples 2, 3, 4, and 5, have similar boiling point distribution patterns and in fact, 4 and 5 appear to be identical. The chemical analyses (Figures 5 and Tables A-4 and A-5) show these materials to be identical.

Sample Nos. 2 and 3 have slightly different patterns, (Figure 2), but overall, the pattern appears to be one of the polyol ester confirmed in Tables A-2 and A-3. Sample No. 2 is high in pentaerithritol ester while Sample No. 3 is high in trimethylol propane ester. Sample No. 7 appears to have a similar pattern to No. 3 in the higher boiling region, but the lower boiling portion might indicate the sample contains diesters or other lower boiling ester material. The chemical analyses of No. 7 reveals its similarity to No. 3 in that it is predominantly trimethylolpropane ester. The fact that no diester material was found in No. 7 indicates that there may be a mixture of two different blends of polyol esters, one, a higher boiling trimethylpropane polyol ester mixture (similar to that in Sample No. 3), and a lower boiling low molecular weight polyol ester mixture. The physical data, especially the viscosity at 40°C, show that No. 3 and No. 7 have physical properties which are different even though the overall chemical composition appear to be the same. However, it must be emphasized that the chemical composition data reflect the component parts which go into the total ester and do not actually represent the composition of the ester in its finished form. In order to adequately define the individual ester molecules, preparative scale separation of the components would have to be performed, followed by hydrolysis and derivatization. Such an approach might involve distillation of the lubricant into narrow cuts followed by analyses of specific cuts for chemical composition. This would be much more time consuming and expensive than the methods currently employed and were beyond the scope of this program. Gas chromatographic/mass spectrographic techniques potentially could also identify the ester type of each peak in Figure 2.

Since Sample Nos. 1, 6, 8, and 9 were mixtures of polyol and dibasic acid esters, high-performance liquid chromatographic separation tech-

niques (gel permeation for molecular size, reverse phase for solubility separation, and absorption for polar separation) were evaluated and found to be ineffective. Fortunately, the polyol and dibasic acid esters in these samples were adequately separated from each other gas chromatographically to allow for quantitation. An emperical approach was also devised based on the major polyol ester and dibasic acid ester alcohols and the direct acid analyses to estimate the basestock ester type percentages given in Tables A-1 through A-10.

Note that Sample Nos. 1 and 6 appear to be identical in Figures 2 and 3, and indeed the chemical composition in Tables A-1 and A-6 supports this conclusion. Sample No. 9 appears in Figure 2 to be similar to Sample Nos. 1 and 6. The chemical analyses in Tables A-1, A-6, and A-9 show that Sample No. 9 is very similar but not identical. Sample No. 10 is unique in the test sample set in that it is the only lubricant containing only diester and no polyol ester.

The daisy graph in Figure 3 aids in the comparison of the chemical composition and complements the boiling point distribution data to help define the lubricants. Note that Sample Nos. 1 and 6 appear to be nearly identical and No. 9 is similar. It can be seen that Sample Nos. 2, 4, and 5 are penta-erithritol-base lubricants and 4 and 5 are identical. Samples 3 and 7 are closely related trimethylolpropane triesters, but there are some differences in the acid distribution. Note that there are more lower molecular weight acids in No. 7 in contrast to heavier acids in No. 3. Sample Nos. 1, 6, 8, and 9 are blends of polyol ester and diesters , while Sample No. 7 is a pure polyol ester and Sample No. 10 is a pure dibasic acid ester, all of which are MIL-L-7808 lubricants.

V. CONCLUSIONS

For this program, four MIL-L-23699B and six MIL-L-7808G turbine engine lubricants have been characterized from the physical properties and chemical composition of their basestocks. The four MIL-L-23699 lubricants were found to be 100-percent polyol esters. Of the six MIL-

L-7808 lubricants, four were mixtures of polyol esters and diesters, one was 100-percent polyol ester, and one was 100-percent diester. Correlation of the chemical data is made to some of the physical properties wherever possible.

VI. REFERENCES

1. L.L. Stavinoha, G.E. Fodor, F.M. Newman, and S.J. Lestz, "Analytical Approach to the Characterization of Military Lubricants," Interim Report AFLRL No. 77, AD A027397, March 1976, Defense Documentation Center, Cameron Station, Alexandria, VA 22314.

2. L.L. Stavinoha, G.E. Fodor, F.M. Newman, and S.J. Lestz, "Analytical Characterization of Synthetic Lubricants," ASLE Transactions, 21, No. 2, p 217-225 (July 1978).

3. L.L. Stavinoha, "Determination of Synthetic Hydrocarbons in Hybrid Mineral Oil Lubricants," Interim Report AFLRL No. 86, AD A039964, January 1977, Defense Documentation Center, Cameron Station, Alexandria, VA, 22314.

4. L.L. Stavinoha, "A Gas Chromatographic Method for Fingerprinting Synthetic Lubricants," Interim Report AFLRL No. 87, MERADCOM Contract No. DAAG53-73-C-0003, June 1977, (Limited Distribution).

5. L.L. Stavinoha, S.J. Lestz, and M.E. LePera, "Synthetic Oil Base Stock and Its Impact on New and Used Engine Oil Analyses," Interim Report AFLRL No. 100, January 1978, MERADCOM Contract No. DAAK70-78-C-0001, Defense Documentation Center, Cameron Station, Alexandria, VA 22314.

6. "Annual Book of ASTM Standards," Parts 23-25 Americal Society of Testing and Materials, Philadelphia, PA.

7. Sniegoski, P.J., "Determination of the Ester Composition of Neopentyl Polyol Ester Lubricants," ASLE Trans., 12:4, 273-279, 1969.

8. "Annual Book of ASTM Standards," Part 24, pp 770-778, 1977, American Society for Testing and Materials, Philadelphia, PA.

APPENDIX

PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR TEN MILITARY TURBINE ENGINE LUBRICANTS

TABLE A-1. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 1

Sample No. 1 AFLRL Code No. AL-7778-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 13.12
  - @ 100°C 3.41
- Viscosity Index 138
- Flash Point, °C(°F) 233 (451)
- Pour Point, °C(°F) -62 (-80)
- API Gravity, @ 15.6°C 18.3

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	330.1	40	426.4	90	468.8
5	402.2	50	427.8	95	484.9
10	418.6	60	430	100	555.3
20	423.6	70	435	Residue = 0	
30	425	80	448		

Chemical Data

- Total Acid No., mg KOH/g 0.12
- Total Base No., mg KOH/g 2.15

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	22.7
Hexanoic, C_6	14.7
iso Heptanoic, C_7	9.1
Heptanoic, C_7	24.5
iso Octanoic, C_8	
Octanoic, C_8	6.1
Nonanoic, C_9	22.9
Decanoic, C_{10}	

- Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic, C_4	
Glutaric, C_5	0.6
Adipic, C_6	1.9
Pimelic, C_7	2.9
Subaric, C_8	4.2
Azelaic, C_9	86.9
Sebacic, C_{10}	1.1
---, C_{11}	2.4
---, C_{12}	

- Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	60
Polyol Ester	40

(Note 1: Identical to #6)

Polyols	wt%
Trimethylolpropane, (TMP)	1.6
Pentaerithritol, (PE)	92
Dipentaerithritol, (DPE)	6.4

Mono Alcohols	
n-Heptanol (C_7)	100
2-Ethylhexanol	
Isooctanol (C_8)	
Isononanol (C_9)	
Isodecanol (C_{10})	
Isoundecanol (C_{11})	
Isododecanol (C_{12})	
Isotridecanol (C_{13})	

TABLE A-2. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 2

Sample No. 2 AFLRL No. AL-7779-L

Physical Data

- Viscosity, cSt,
  - @ 40°C: 24.99
  - @ 100°C: 5.17
- Viscosity Index: 142
- Flash Point, °C(°F): 263(505)
- Pour Point, °C(°F): -48(-54)
- API Gravity, @ 15.6°C: *

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	372.2	40	466	90	505.6
5	422.9	50	470.2	95	522
10	436.5	60	477.9	100	555.3
20	449.4	70	484.2	Residue = 0	
30	455.9	80	493.3		

Chemical Data

- Total Acid No., mg KOH/g: *
- Total Base No., mg KOH/g: *

Basestock Type	wt%
Dibasic Acid Ester	0
Polyol Ester	100

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	1
Pentanoic, C_5	33.7
Hexanoic, C_6	0.5
iso Heptanoic, C_7	
Heptanoic, C_7	33.3
iso Octanoic, C_8	3.3
Octanoic, C_8	3
Nonanoic, C_9	25.3
Decanoic, C_{10}	

Polyols	wt%
Trimethylolpropane, (TMP)	3.2
Pentaerithritol, (PE)	87.2
Dipentaerithritol, (DPE)	9.5

- Dibasic Acid Ester Components

Dicarboxylic Acids
Succinic, C_4
Glutaric, C_5
Adipic, C_6
Pimelic, C_7
Subaric, C_8
Azelaic, C_9
Sebacic, C_{10}
---, C_{11}
---, C_{12}

Mono Alcohols
- n-Heptanol (C_7)
- 2-Ethylhexanol
- Isooctanol (C_8)
- Isononanol (C_9)
- Isodecanol (C_{10})
- Isoundecanol (C_{11})
- Isododecanol (C_{12})
- Isotridecanol (C_{13})

* Insufficient sample

TABLE A-3. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 3

Sample No. 3 AFLRL No. AL-7780-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 23.84
  - @ 100°C 4.99
- Viscosity Index 140
- Flash Point, °C(°F) 249 (481)
- Pour Point, °C(°F) -58 (-72)
- API Gravity, @ 15.6°C 13.4

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	362.9	40	460.2	90	518.6
5	422.1	50	466.7	95	539
10	436.5	60	470.9	100	555.3
20	446.5	70	477.2	Residue = 0	
30	453.7	80	487.7		

Chemical Data

- Total Acid No., mg KOH/g 0.6
- Total Base No., mg KOH/g 2.09

- Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	0
Polyol Ester	100

- Polyo. Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	19.6
Hexanoic, C_6	2.6
iso Heptanoic, C_7	2.5
Heptanoic, C_7	22.6
iso Octanoic, C_8	
Octanoic, C_8	30.4
Nonanoic, C_9	0.6
Decanoic, C_{10}	21.7

Polyols	wt%
Trimethylolpropane, (TMP)	92.9
Pentaerithritol, (PE)	6.0
Dipentaerithritol, (DPE)	1.1

- Dibasic Acid Ester Components

Dicarboxylic Acids
- Succinic, C_4
- Glutaric, C_5
- Adipic, C_6
- Pimelic, C_7
- Subaric, C_8
- Azelaic, C_9
- Sebacic, C_{10}
- ---, C_{11}
- ---, C_{12}

Mono Alcohols
- n-Heptanol (C_7)
- 2-Ethylhexanol
- Isooctanol (C_8)
- Isononanol (C_9)
- Isodecanol (C_{10})
- Isoundecanol (C_{11})
- Isododecanol (C_{12})
- Isotridecanol (C_{13})

TABLE A-4. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 4

Sample No. 4 AFLRL No. AL-7781-L

Physical Data

- Viscosity, cSt,
  - @ 40°C: 24.70
  - @ 100°C: 4.98
- Viscosity Index: 130
- Flash Point, °C(°F): 256(493)
- Pour Point, °C(°F): -43(-45)
- API Gravity, @ 15.6°C: *

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	364.3	40	463.1	90	511.1
5	420	50	471.6	95	528.8
10	432.1	60	477.9	100	555.3
20	446.5	70	486.3	Residue = 0	
30	455.9	80	495.4		

Chemical Data

- Total Acid No., mg KOH/g: *
- Total Base No., mg KOH/g: *

- Basestock Type
  - Dibasic Acid Ester: 0
  - Polyol Ester (Identical to #5): 100

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	11.3
Pentanoic, C_5	21.6
Hexanoic, C_6	1.5
iso Heptanoic, C_7	
Heptanoic, C_7	31.2
iso Octanoic, C_8	
Octanoic, C_8	20.1
Nonanoic, C_9	
Decanoic, C_{10}	14.2

Polyols	wt%
Trimethylolpropane, (TMP)	1.3
Pentaerithritol, (PE)	91.2
Dipentaerithritol, (DPE)	7.5

- Dibasic Acid Ester Components

Dicarboxylic Acids
- Succinic, C_4
- Glutaric, C_5
- Adipic, C_6
- Pimelic, C_7
- Subaric, C_8
- Azelaic, C_9
- Sebacic, C_{10}
- ---, C_{11}
- ---, C_{12}

Mono Alcohols
- n-Heptanol (C_7)
- 2-Ethylhexanol
- Isooctanol (C_8)
- Isononanol (C_9)
- Isodecanol (C_{10})
- Isoundecanol (C_{11})
- Isododecanol (C_{12})
- Isotridecanol (C_{13})

* Insufficient Sample

TABLE A-5. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 5

Sample No. 5 AFLRL No. AL-7782-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 25.03
  - @ 100°C 5.00
- Viscosity Index 128
- Flash Point, °C(°F) 254(490)
- Pour Point, °C(°F) -48(-54)
- API Gravity, @ 15.6°C *

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	364.3	40	463.1	90	509.7
5	419.3	50	470.9	95	526.7
10	430.7	60	477.9	100	555.3
20	445.1	70	485.6	Residue = 0	
30	455.2	80	494.7		

Chemical Data

- Total Acid No., mg KOH/g *
- Total Base No., mg KOH/g *

- Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	0
Polyol Ester (Identical to #4)	100

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	11.3
Pentanoic, C_5	21.1
Hexanoic, C_6	1.4
iso Heptanoic, C_7	
Heptanoic, C_7	29.4
iso Octanoic, C_8	3.0
Octanoic, C_8	19.2
Nonanoic, C_9	
Decanoic, C_{10}	14.6

Polyols	wt%
Trimethylolpropane, (TMP)	1.3
Pentaerithritol, (PE)	93.4
Dipentaerithritol, (DPE)	5.3

- Dibasic Acid Ester Components

Dicarboxylic Acids
Succinic, C_4
Glutaric, C_5
Adipic, C_6
Pimelic, C_7
Subaric, C_8
Azelaic, C_9
Sebacic, C_{10}
---, C_{11}
---, C_{12}

Mono Alcohols
n-Heptanol (C_7)
2-Ethylhexanol
Isooctanol (C_8)
Isononanol (C_9)
Isodecanol (C_{10})
Isoundecanol (C_{11})
Isododecanol (C_{12})
Isotridecanol (C_{13})

* Insufficient Sample

TABLE A-6. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 6

Sample No. 6 AFLRL No. AL-7783-L

Physical Data

- Viscosity, cSt,
  - @ 40°C: 12.96
  - @ 100°C: 3.35
- Viscosity Index: 135
- Flash Point, °C(°F): 228 (441)
- Pour Point, °C(°F): -61 (-78)
- API Gravity, @ 15.6°C: 18.5

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	364.3	40	424.3	90	473.7
5	400	50	425.7	95	491.9
10	415	60	427.1	100	555.3
20	421.4	70	432.1	Residue = 0	
30	422.9	80	448		

Chemical Data

- Total Acid No., mg KOH/g: 0.08
- Total Base No., mg KOH/g: 2.03

- Basestock Type
  - Dibasic Acid Ester: 60 wt%
  - Polyol Ester (Identical to #1): 40 wt%

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	14.7
Hexanoic, C_6	10.1
iso Heptanoic, C_7	9.0
Heptanoic, C_7	34.4
iso Octanoic, C_8	
Octanoic, C_8	4.6
Nonanoic, C_9	26.3
Decanoic, C_{10}	0.9

Polyols	wt%
Trimethylolpropane, (TMP)	3.7
Pentaerithritol, (PE)	89.7
Dipentaerithritol, (DPE)	6.6

- Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic, C_4	
Glutaric, C_5	0.3
Adipic, C_6	2.9
Pimelic, C_7	3.1
Subaric, C_8	4.3
Azelaic, C_9	83.9
Sebacic, C_{10}	1.7
---, C_{11}	3.7
---, C_{12}	

Mono Alcohols	
n-Heptanol (C_7)	100
2-Ethylhexanol	
Isooctanol (C_8)	
Isononanol (C_9)	
Isodecanol (C_{10})	
Isoundecanol (C_{11})	
Isododecanol (C_{12})	
Isotridecanol (C_{13})	

TABLE A-7. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 7

Sample No. 7 AFLRL No. AL-7784-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 12.58
  - @ 100°C 3.28
- Viscosity Index 134
- o Flash Point, °C(°F) 211 (411)
- o Pour Point, °C(°F) -67 (-89)
- o API Gravity, @ 15.6°C 16.7

- Boiling Point Distribution (Normalization to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	355	40	407.9	90	475.1
5	377.9	50	420	95	484.2
10	386.5	60	435	100	555.3
20	397.9	70	448.7	Residue = 0	
30	401.5	80	461.7		

Chemical Data

- Total Acid No., mg KOH/g 0.21
- Total Base No., mg KOH/g 1.36

- Basestock Type
  - Dibasic Acid Ester 0
  - Polyol Ester 100

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	10.8
Hexanoic, C_6	1.8
iso Heptanoic, C_7	
Heptanoic, C_7	29.2
iso Octanoic, C_8	
Octanoic, C_8	24.2
Nonanoic, C_9	16.7
Decanoic, C_{10}	17.3

Polyols	wt%
Trimethylolpropane, (TMP)	100
Pentaerithritol, (PE)	
Dipentaerithritol, (DPE)	

- Dibasic Acid Ester Components

Dicarboxylic Acids
Succinic, C_4
Glutaric, C_5
Adipic, C_6
Pimelic, C_7
Subaric, C_8
Azelaic, C_9
Sebacic, C_{10}
---, C_{11}
---, C_{12}

Mono Alcohols
- n-Heptanol (C_7)
- 2-Ethylhexanol
- Isooctanol (C_8)
- Isononanol (C_9)
- Isodecanol (C_{10})
- Isoundecanol (C_{11})
- Isododecanol (C_{12})
- Isotridecanol (C_{13})

TABLE A-8. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 8

Sample No. 8 AFLRL No. AL-7785-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 13.34
  - @ 100°C 3.47
- Viscosity Index 144
- Flash Point, °C(°F) 216 (421)
- Pour Point, °C(°F) -66 (-87)
- API Gravity, @ 15.6°C 16.4

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	385	40	400.7	90	442.2
5	389.3	50	435.7	95	442.9
10	390	60	437.9	100	555.3
20	391.5	70	439.3	Residue = 0	
30	393.6	80	440.8		

Chemical Data

- Total Acid No., mg KOH/g 0.33
- Total Base No., mg KOH/g 1.29

- Basestock Type
  - Dibasic Acid Ester 40 wt%
  - Polyol Ester 60 wt%

- Polyol Ester Components

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	
iso Hexanoic, C_6	7.5
Hexanoic, C_6	0.7
iso Heptanoic, C_7	
Heptanoic, C_7	89.4
iso Octanoic, C_8	
Octanoic, C_8	
Nonanoic, C_9	2.4
iso Decanoic, C_{10}	
Decanoic, C_{10}	

Polyols	wt%
Trimethylolpropane, (TMP)	100
Pentaerithritol, (PE)	
Dipentaerithritol, (DPE)	

- Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic, C_4	
Glutaric, C_5	
Adipic, C_6	86.1
Pimelic, C_7	1.3
Subaric, C_8	
Azelaic, C_9	3.8
Sebacic, C_{10}	8.9
---, C_{11}	
---, C_{12}	

Mono Alcohols	
n-Heptanol (C_7)	100
2-Ethylhexanol	
Isooctanol (C_8)	
Isononanol (C_9)	
Isodecanol (C_{10})	
Isoundecanol (C_{11})	
Isododecanol (C_{12})	
Isotridecanol (C_{13})	

TABLE 9. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPE NO. 9

Sample No. 9 AFLRL No. AL-7786-L

Physical Data

- Viscosity, cSt,
  - @ 40°C 9.17
  - @ 100°C 2.74
- Viscosity Index 150
- Flash Point, °C(°F) 116 (241)
- Pour Point, °C(°F) -67 (-89)
- API Gravity, @ 15.6°C 21.4

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	367.2	40	424.3	90	477.9
5	400	50	425.7	95	496.8
10	415	60	427.1	100	555.3
20	421.4	70	435.7	Residue = 0	
30	422.9	80	449.4		

Chemical Data

- Total Acid No., mg KOH/g 0.07
- Total Base No., mg KOH/g 1.78

- Basestock Type

Basestock Type	wt%
Dibasic Acid Ester	50
Polyl Ester	50

- *Polyol Ester Components*

Monocarboxylic Acids	wt%
Butanoic, C_4	
iso Pentanoic, C_5	
Pentanoic, C_5	28.3
Hexanoic, C_6	5.7
iso Heptanoic, C_7	12.6
Heptanoic, C_7	29.6
iso Octanoic, C_8	
Octanoic, C_8	4.1
Nonanoic, C_9	19.6
Decanoic, C_{10}	

Polyols	wt%
Trimethylolpropane, (TMP)	9.2
Pentaerithritol, (PE)	84.7
Dipentaerithritol, (DPE)	6.1

- Dibasic Acid Ester Components

Dicarboxylic Acids	
Succinic, C_4	
Glutaric, C_5	
Adipic, C_6	1.3
Pimelic, C_7	2.2
Subaric, C_8	10.7
Azelaic, C_9	81.9
Sebacic, C_{10}	1.3
---, C_{11}	2.6
---, C_{12}	

Mono Alcohols	
n-Heptanol (C_7)	100
2-Ethylhexanol	
Isooctanol (C_8)	
Isononanol (C_9)	
Isodecanol (C_{10})	
Isoundecanol (C_{11})	
Isododecanol (C_{12})	
Isotridecanol (C_{13})	

TABLE A-10. PHYSICAL AND CHEMICAL CHARACTERIZATION DATA FOR SAMPLE NO. 10

Sample No. 10 AFLRL No. AL-7787-L

Physical Data

- Viscosity, cSt,
  - @ 40°C: 12.76
  - @ 100°C: 3.54
- Viscosity Index: 164
- Flash Point, °C(°F): 194 (381)
- Pour Point, °C(°F): -68 (-90)
- API Gravity, @ 15.6°C: 20.5

- Boiling Point Distribution (Normalized to 100%)

wt% off	°C	wt% off	°C	wt% off	°C
0.5	375.7	40	397.2	90	432.9
5	385	50	399.3	95	440.8
10	387.9	60	402.2	100	555.3
20	392.2	70	419.3	Residue = 0	
30	395	80	427.8		

Chemical Data

- Total Acid No., mg KOH/g: 0.37
- Total Base No., mg KOH/g: 3.37

- Basestock Type
  - Dibasic Acid Ester: 100 wt%
  - Polyol Ester: 0

- Polyol Ester Components

Monocarboxylic Acids	wt%	Polyols	wt%
Butanoic, C_4			
iso Pentanoic, C_5		Trimethylolpropane, (TMP)	
Pentanoic, C_5			
Hexanoic, C_6			
iso Heptanoic, C_7		Pentaerithritol, (PE)	
Heptanoic, C_7			
iso Octanoic, C_8		Dipentaerithritol, (DPE)	
Octanoic, C_8			
Nonanoic, C_9			
Decanoic, C_{10}			

- Dibasic Acid Ester Components

Dicarboxylic Acids	wt%	Mono Alcohols	wt%
Succinic, C_4		n-Heptanol (C_7)	
Glutaric, C_5		2-Ethylhexanol	
Adipic, C_6	98.1	Isooctanol (C_8)	64
Pimelic, C_7		Isononanol (C_9)	
Subaric, C_8		Isodecanol (C_{10})	36
Azelaic, C_9	1.6	Isoundecanol (C_{11})	
Sebacic, C_{10}	0.3	Isododecanol (C_{12})	
---, C_{11}		Isotridecanol (C_{13})	
---, C_{12}			

DISTRIBUTION

Commander U.S. Army Mobility Equipment Research & Development Command Attn: DRDME-GL, Mr. M.E. LePera Fort Belvoir, VA 22060	10
DCASMA, San Antonio P.O. Box 1040 Main Govt Office 615 East Houston St. San Antonio, TX 78294	2
Defense Documentation Center Cameron Station Alexandria, VA 22314	12
Mr. Edward J. Hollman (DRDAV-Q) Director of Product Assurance U.S. Army Aviation Research & Development Command P.O.Box 209 St. Louis, MO 63166	10
Dr. Erwin V. Zaretsky Bearying, Gearing, and Transmission Section NASA/Lewis Research Center 21000 Brookpark Road Cleveland, OH 44135	10